IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of: Shinichi UDA et al.	
Application No.: 10/583,564	Confirmation No.: 8975
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For: METHOD FOR PRODUCING WATER- ABSORBING RESIN	Examiner: HUHN, Richard A

DECLARATION UNDER 37 C.F.R. 1.132

COMMISSIONER FOR PATENTS
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Sir:

I, Tomoki KAWAKITA, residing in Hyogo-ken, Japan, hereby declare and state as follows:

1.   That I am a co-inventor of the above-identified application, and thoroughly familiar with the contents of U.S. Application Serial No. 10/583,564 filed on June 19, 2006, entitled METHOD FOR PRODUCING WATER-ABSORBING RESIN, its prosecution before the United States Patent and Trademark Office and the references cited therein.

2.   I am a graduate from Nagoya Institute of Technology, faculty of applied chemistry and received a bachelor's degree in the year 1991, majoring in applied chemistry.

3.   I have been employed in Sumitomo Seika Chemicals Co., Ltd. in the year 1991 and have been assigned to the Research Laboratories.

4.   I have been involved in the research and development of water-absorbent resin on the years 2002-2003 and 2007-2008.

5.   The following experiments were conducted by myself or under my direct supervision and control in order to verify that the water-absorbent resin obtained in the present invention is distinguishable from Japanese Patent Laid-Open No. Hei 09-012613 (simply referred to as JP '613).

EXPERIMENTAL METHOD
PURPOSE OF EXPERIMENT

The water-absorbent resins of Examples 1 to 15 of JP '613 are obtained by a method including the step of subjecting water-soluble ethylenically unsaturated monomers to a reversed phase suspension polymerization in two steps, and in both the first-step and second-step polymerizations, the monomers are subjected to the reversed phase suspension polymerization in the presence of a phosphorus-containing compound.

In addition, the water-absorbent resin of Comparative Example 2 of the present application is obtained by a method including the step of subjecting water-soluble ethylenically unsaturated monomers to the reversed phase suspension polymerization in two steps, in which the monomers are subjected to the reversed phase suspension polymerization in the presence of a phosphorus-containing compound only in the first-step polymerization.

Therefore, for the purpose of this experiment, it is clarified that in a method for producing a water-absorbent resin including the step of subjecting the monomers to a reversed phase suspension polymerization in at least two steps, the monomers are subjected to the reversed phase suspension polymerization in the presence of a phosphorus-containing compound in the first-step polymerization, whereby a water-absorbent resin having a fast water absorption rate and a low amount of water-soluble substance as taught in the present invention cannot be obtained.

SYNTHESIS OF WATER-ABSORBENT RESIN

The amount 340 g of n-heptane and 0.92 g of a sucrose fatty acid ester (manufactured by MITSUBISHI CHEMICAL CORPORATION under the trade name of S-370) were added to a 1000 mL-five-necked cylindrical round bottomed flask equipped with a stirrer, a reflux condenser, a dropping funnel, a thermometer and a nitrogen gas inlet tube. The mixture was dispersed in the flask, and the temperature of the dispersion was raised to dissolve the mixture, and thereafter the resulting solution was cooled to 55°C.

Separately from the above, 92 g of an 80% by weight aqueous solution of acrylic acid (1.02 mol) was added to a 500 mL-Erlenmeyer flask. Thereto was added dropwise 102.2 g of a 30% by weight aqueous sodium hydroxide (0.77 mol) with cooling from external, to neutralize 75% by mol of acrylic acid. Further, 50.2 g of water, 0.11 g (0.00041 mol) of potassium persulfate, 8.3 mg (0.000047 mol) of ethylene glycol diglycidyl ether and 0.41 g (0.0019 mol) of

disodium phosphite pentahydrate were added thereto, to give an aqueous

monomer solution for a first-step polymerization.

The entire amount of this aqueous monomer solution for a first-step

polymerization was added to the above-mentioned round bottomed flask with

stirring, and the mixture was dispersed. After the internal of the system was

sufficiently replaced with nitrogen gas, the temperature of the content mixture

was raised to 70°C, and the polymerization reaction was carried out for 1 hour

while keeping its bath temperature at 70°C. Thereafter, the resulting reaction

mixture in the form of a slurry was cooled to room temperature.

Separately from the above, 119.1 g of an 80% by weight aqueous solution

of acrylic acid (1.32 mol) was added to a separate 500 mL-Erlenmeyer flask.

The amount 132.2 g of a 30% by weight aqueous sodium hydroxide (0.99 mol)

was added dropwise thereto while cooling, to neutralize 75% by mol of acrylic

acid. Further, 27.4 g of water, 0.14 g (0.00052 mol) of potassium persulfate and

0.54 g (0.0025 mol) of disodium phosphite pentahydrate were added thereto, to

give an aqueous monomer solution for a second-step polymerization. The

aqueous monomer solution was cooled in an ice water bath.

The entire amount of this aqueous monomer solution for a second-step

polymerization was added to the reaction mixture in the form of a slurry obtained

above. Thereafter, the internal of the system was again sufficiently replaced with

nitrogen gas, the temperature of the reaction mixture was then raised to 70°C,

and the second-step polymerization reaction was carried out for 2 hours while

keeping its bath temperature at 70°C. After the termination of the

polymerization, the polymerization slurry was heated in an oil bath at 120°C, and

the heated mixture was subjected to azeotropic distillation to distill off 257 g of water to external of the system. The amount of remaining water in the reaction system at this point was 61 g.

Next, 7.81 g of a 2% by weight aqueous solution of ethylene glycol diglycidyl ether (0.00090 mol) was added to the flask while mixing, and a post-crosslinking treatment was carried out. Water and n-heptane were further distilled off from the mixture by distillation, and the residue was dried, to give 223.0 g of a water-absorbent resin A having a mass-average particle size of 370 μm.

METHODS FOR DETERMINING PROPERTIES OF WATER-ABSORBENT RESIN

The water-absorbent resin A was evaluated for the water absorption rate and the amount of water-soluble substance in accordance with the following methods.

(1)   Water Absorption Rate

The amount 50 ± 0.01 g of physiological saline at a temperature of 23° to 26°C was weighed out in a 100 mL beaker. A magnetic stirrer bar having a size of 8 mmφ × 30 mm without a ring was placed in the beaker, and the beaker was placed on the top of MAGNETIC STIRRER (manufactured by IUCHI under the product number of HS-30D). Subsequently, the magnetic stirrer bar was adjusted so that the magnetic stirrer bar rotated at 600 rpm, and further adjusted

so that the bottom of the vortex generated by the rotation of the magnetic stirrer bar came near the upper portion of the magnetic stirrer bar.

Next, 1.0 ± 0.002 g of particles obtained by sieving the water-absorbent resin with two kinds of standard sieve complying with JIS-Z8801 (1982) (opening of sieve: 500 μm or 300 μm), and subjecting the sieved water-absorbent resin to a particle size adjustment (500 μm or less and 300 μm or more) were quickly poured between the center of vortex in the beaker and the side of the beaker, and the time (seconds) from a point where the water-absorbent resin was poured into the beaker to a point where the vortex converged was determined with a stopwatch, which is defined as a water absorption rate.

(2)   Amount of Water-Soluble Substance

The amount 500 ± 0.1 g of physiological saline was weighed out in a 500 mL beaker. A magnetic stirrer bar having a size of 8 mmφ × 30 mm without a ring was placed in the beaker, and the beaker was placed on the top of MAGNETIC STIRRER (manufactured by IUCHI under the product number of HS-30D). Subsequently, the magnetic stirrer bar was adjusted so that the magnetic stirrer bar rotated at 600 rpm, and further adjusted so that the bottom of the vortex generated by the rotation of the magnetic stirrer bar came near the upper portion of the magnetic stirrer bar.

Next, 2.0 ± 0.002 g of particles obtained by sieving the water-absorbent resin with two kinds of standard sieve complying with JIS-Z8801-1982 (opening of sieve: 500 μm or 300 μm), and subjecting the sieved water-absorbent resin to a particle size adjustment (500 μm or less and 300 μm or more) were quickly

poured between the center of vortex in the beaker and the side of the beaker and dispersed therein, and the mixture was stirred for 3 hours. The aqueous dispersion of the water-absorbent resin after stirring for 3 hours was filtered with a standard sieve (opening of sieve: 75 μm), and the filtrate obtained was further subjected to suction filtration using a Kiriyama type funnel (Filter Paper No. 6).

The amount 80 ± 0.1 g of the filtrate obtained was weighed out in a 100 mL beaker dried beforehand to a constant weight, and the filtrate was dried with a forced convection oven (manufactured by ADVANTEC) at 140°C until a constant weight was attained. A weight W_d (g) of the solid content of the filtrate was determined.

On the other hand, the same procedures as the above were carried out without using the water-absorbent resin, and a weight W_e (g) of the solid content of the filtrate was determined. The amount of water-soluble substance was calculated on the basis of the following formula:

Amount of Water-Soluble Substance (% by weight)
$$= [[(W_d - W_e) \times (500/80)]/2] \times 100 .$$

DETERMINATION RESULTS

The determination results are shown in the following table.

		Addition of Phosphorus-Containing Compound		Water Absorption Rate	Amount of Water-Soluble Substance
		1st Step	2nd Step	(sec)	(% by weight)
Present Invention	Ex. 1	-	Added	41	16
	Comp. Ex. 2	Added	-	70	35
Water-Absorbent Resin A		Added	Added	69	39

It has been verified that in the method for producing a water-absorbent resin including the step of subjecting the monomers to multi-steps of at least two steps of the reversed phase suspension polymerization, when the monomers are subjected to a reversed phase suspension polymerization in the presence of a phosphorus-containing compound in the first-step polymerization, the resulting water-absorbent resin A and the water-absorbent resin of Comparative Example 2 had slower water absorption rate and higher amount of water-soluble substance, as compared to the water-absorbent resin of the present invention.

Statement Under 18 U.S.C. § 1001

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dated: _____   By _____

Tomoki KAWAKITA